

with the mean value of the ring torsion angles being  $0.5 (5)^\circ$ .

The piroxicam molecule is not far from being planar ( $\sim bc$  plane); an amide group is involved in an intramolecular hydrogen bond to the hydroxy group,  $O(17)-H(17)\cdots O(15)$   $2.561 (3) \text{ \AA}$ ; the torsion angles along bonds connecting this part of the molecule are  $C(3)-C(4)-C(10)-C(5)$   $159.8 (3)$  and  $C(3)-C(14)-N(16)-C(2')$   $177.9 (3)^\circ$ .  $S(1)$  and  $N(2)$  with non-ring atoms bonded to them show significant departure from the plane of the molecule. The intermolecular hydrogen bond between the amido N and the sulphoxide group [ $N(16)-H(16)\cdots O(11)$   $3.053 (3) \text{ \AA}$ ] connects piroxicam molecules in an infinite chain along  $b$ .

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## 2-Amino-5,5-dimethyl-1-pyrrolinium Diethyl Phosphate Monohydrate

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**Abstract.**  $C_6H_{13}N_2^+ \cdot C_4H_{10}O_4P^- \cdot H_2O$ , monoclinic,  $P2_1/c$ ,  $a = 14.138 (8)$ ,  $b = 9.325 (7)$ ,  $c = 12.369 (8) \text{ \AA}$ ,  $\beta = 107.71 (5)^\circ$ ,  $V = 1553.4 \text{ \AA}^3$  at 295 K, with  $Z = 4$ ,  $D_m = 1.21$  (floatation),  $D_x = 1.215 \text{ g cm}^{-3}$ . The structure was solved by direct methods and refined by full-matrix least squares to a final weighted  $R$  factor of 0.083 and conventional  $R$  factor of 0.072, based on 1858 reflections with significant intensity. The structure involves four modes of hydrogen bonding between amidinium and phosphate ions, two of which involve water acting as a bridge. The  $O(\text{ethyl})-P-O-C$  torsion angles are  $\pm 67.1 (6)$  and  $\pm 63.3 (6)^\circ$ .

**Introduction.** 2-Amino-5,5-dimethylpyrrolinium diethyl phosphate was synthesized as a model for protein-nucleotide interactions by Dr D. A. Usher, De-

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partment of Chemistry, Cornell University, who provided the crystals for this study. The Laue symmetry and systematic absences ( $h0l$ ,  $l = 2n + 1$ ;  $0k0$ ,  $k = 2n + 1$ ) identified the space group as  $P2_1/c$ . The unit-cell dimensions were refined by least squares from observations of 12 reflections made on a FACS-I automated diffractometer, which was also used for data collection.

Data were collected with monochromatized  $Mo K\alpha$  radiation in the  $\theta-2\theta$  scan mode between  $3$  and  $48^\circ$ , with a minimum base width of  $2^\circ$ , a scan rate of  $2^\circ \text{ min}^{-1}$  and 40 s background counts. The crystal was of irregular shape and dimensions  $0.5 \times 0.5 \times 0.3 \text{ mm}$ . The intensities of the 2488 independent reflections were corrected for absorption assuming a spherical crystal, with  $\mu(Mo K\alpha) = 0.197 \text{ mm}^{-1}$ .

The resulting structure factors were scaled by the Wilson statistical method, and normalized structure factors were calculated. These were then used in the direct-method programs of Germain & Woolfson (1968). An  $E$  map calculated using the best-non-trivial solution yielded coordinates for all non-H atoms of the

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Table 1. *Structural parameters*

(a) Positional coordinates ( $\times 10^4$ ) of the non-H atoms\* and their equivalent isotropic thermal parameters ( $\times 10^3$ ) with e.s.d.'s in parentheses.

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$ ( $\text{\AA}^2$ )†
P	2417 (1)	1264 (1)	7658 (1)	52 (1)
O(1)	1623 (2)	171 (3)	6912 (2)	65 (1)
O(2)	1896 (2)	2780 (3)	7388 (2)	71 (1)
O(3)	2570 (2)	996 (3)	8883 (2)	64 (1)
O(4)	3278 (2)	1180 (3)	7222 (2)	63 (1)
O(5)	3954 (2)	2717 (3)	5684 (3)	65 (2)
N(1)	6475 (2)	3488 (4)	5000 (3)	57 (2)
N(2)	5153 (3)	4573 (4)	3663 (3)	58 (2)
C(1)	5638 (3)	3457 (4)	4187 (3)	50 (2)
C(2)	5294 (4)	1959 (4)	3935 (4)	65 (2)
C(3)	6147 (4)	1068 (5)	4610 (5)	113 (3)
C(4)	6834 (3)	2075 (4)	5488 (3)	66 (2)
C(5)	7902 (5)	1881 (9)	5554 (7)	115 (4)
C(6)	6653 (8)	1982 (10)	6623 (6)	115 (5)
C(7)	689 (3)	-9 (6)	7124 (5)	81 (3)
C(8)	141 (5)	-1232 (7)	6438 (5)	100 (3)
C(9)	1704 (6)	3363 (7)	6243 (4)	134 (4)
C(10)	1224 (7)	4590 (8)	6077 (5)	171 (6)

(b) H-atom coordinates ( $\times 10^3$ ) and isotropic thermal parameters ( $\times 10^2$ ) with e.s.d.'s where known.

	<i>x</i>	<i>y</i>	<i>z</i>	$U$ ( $\text{\AA}^2$ )
H(O5)1	351 (4)	291 (5)	511 (4)	9 (2)
H(O5)2	367 (3)	219 (5)	605 (4)	7 (1)
H(N1)	670 (3)	418 (4)	525 (3)	5 (1)
H(N2)1	453 (3)	444 (4)	314 (3)	7 (1)
H(N2)2	550 (3)	551 (5)	392 (3)	8 (1)
H(C2)1	469 (4)	193 (6)	414 (4)	11 (2)
H(C2)2	514 (4)	181 (6)	314 (5)	13 (2)
H(C3)1	651	65	410	12
H(C3)2	591	27	500	12
H(C5)1	821	261	615	11
H(C5)2	812 (4)	107 (6)	583 (5)	12 (2)
H(C5)3	808 (4)	207 (6)	500 (5)	11 (2)
H(C6)1	594	228	645	11
H(C6)2	706 (4)	247 (7)	704 (5)	10 (3)
H(C6)3	678 (5)	118 (7)	679 (5)	12 (2)
H(C7)1	39 (5)	92 (7)	683 (5)	14 (2)
H(C7)2	72 (3)	-8 (5)	796 (4)	10 (2)
H(C8)1	-28	-87	568	18
H(C8)2	55 (5)	-186 (9)	634 (7)	18 (3)
H(C8)3	-30	-169	684	18

\* The crystallographic numbering sequence is not the same as the chemical numbering sequence assumed in the title.

$$\dagger U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

charged species. A difference synthesis revealed the O atom of the water molecule, O(5). Further refinement using the program *ORFLS* (Busing, Martin & Levy, 1962) and difference synthesis revealed 15 of the 25 H atoms.

The 1858 reflections for which  $|F_o| > 3.5\sigma(|F_o|)$ , where  $\sigma(|F_o|)$  was estimated as  $\sigma'(|F_o|) + 1.0 + 0.1|F_o|$  and  $\sigma'(|F_o|)$  was based on counting statistics, were used in the refinement.

Further refinement was then carried out with the program *CUDLS*, written by J. S. Stephens, McMaster University, Hamilton, Ontario, Canada. This program allowed correction for secondary extinction using the expression  $F' = F(1 + gF^2Lp)$  where  $Lp$  is the Lorentz and polarization correction (Stout & Jensen, 1968). The factor  $g$  refined to the final value of  $3.2 \times 10^{-4}$ . Refinement was then continued using the program *ORFLS*. Five additional H atoms were placed assuming  $sp^3$  symmetry. The temperature factors of these atoms were assigned values calculated from the average least-squared temperature factors of similar H atoms. Neither the positions nor the temperature factors of these H atoms were refined. The electron density in the vicinity of the ethyl group on the atom O(2), and the refined bond lengths of this group suggested that it was disordered. No attempt was made to place the five H atoms associated with it.

The weighting scheme used in the final cycles of least squares  $[w(F_o) = (0.717 - 0.0497|F_o| + 0.00272|F_o|^2 - 0.000013|F_o|^3)^{-2}]$  was based on the assumption that the average value of  $||F_o| - |F_c||$  in a range of  $|F_o|$  is the best available estimate of  $\sigma(|F_o|)$ . The scattering factors for the non-H atoms were taken from Cromer & Mann (1968). The scattering factors for the H atoms were taken from Stewart, Davidson & Simpson (1965). The final values of the conventional  $R$  factor and the weighted  $R$  factor,  $R_w$   $\{R_w = [\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2]^{1/2}\}$  were 0.072 and 0.083. All shifts in the final cycles of least squares were less than 25% of their e.s.d.'s.\* The final atomic

\* Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36975 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Hydrogen-bonding geometry*

<i>D-H...A</i>	<i>D-H</i>	<i>H...A</i>	<i>D...A</i>	<i>D-H...A</i>
O(5)-H(O5)2...O(4)	0.84 (5) Å	1.95 (5) Å	2.773 (5) Å	166 (4)°
O(5)-H(O5)1...O(3)	0.81 (5)	1.97 (5)	2.754 (5)	162 (4)
N(1)-H(N1)...O(3)	0.75 (4)	2.10 (4)	2.840 (5)	173 (3)
N(2)-H(N2)1...O(4)	0.93 (5)	1.88 (4)	2.797 (5)	169 (3)
N(2)-H(N2)2...O(5)	1.01 (5)	1.83 (6)	2.830 (6)	176 (4)

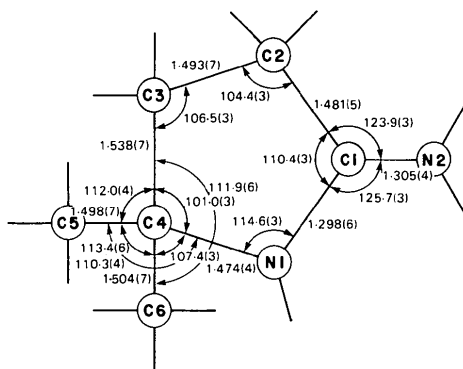


Fig. 1. Atom labelling, bond lengths (Å) and bond angles ( $^{\circ}$ ) with e.s.d.'s for the 2-amino-5,5-dimethyl-1-pyrrolinium cation.

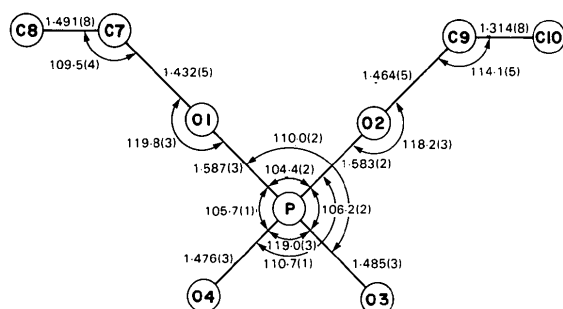


Fig. 2. Atom labelling, bond lengths (Å) and bond angles ( $^{\circ}$ ) with e.s.d.'s for the diethyl phosphate anion.

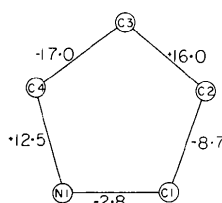


Fig. 3. Endocyclic torsion angles ( $^{\circ}$ ) for the five-membered ring. E.s.d.'s are 0.6 $^{\circ}$ .

coordinates are given in Table 1 with e.s.d.'s. The atom-labelling sequence, bond lengths and bond angles for the charged species are given in Figs. 1 and 2. The geometry of the hydrogen bonds is described in Table 2. The endocyclic torsional angles in the five-membered ring are given in Fig. 3.

**Discussion.** The packing of the three molecules is shown in Fig. 4 which was prepared with the aid of the program *ORTEP* (Johnson, 1965). The structure consists of layers stacked normal to the *a* axis. The outer surfaces of a layer are formed by the ethyl and methyl moieties of the anion and cation respectively. The water molecule, the N atoms and the unsubstituted phosphate O atoms are nearer the center of the

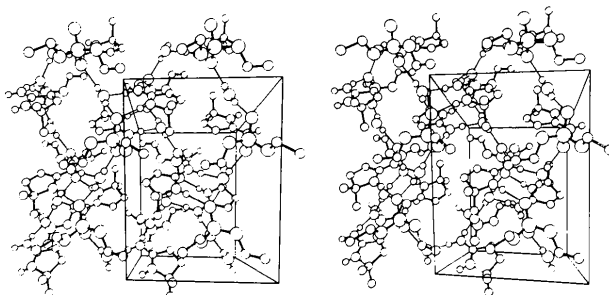


Fig. 4. Stereoscopic view of the packing of the unit cell. The *a* axis points out of the page, the *b* axis is horizontal and the *c* axis is vertical.

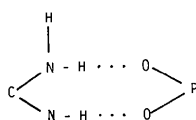
layer. These atoms form five hydrogen bonds, two making direct linkages between the amidinium ion and the phosphate ion. The others create two linkages between the charged species with the water molecule acting as a bridge.

The pattern of the torsional angles for the amidinium ion is not readily identifiable as showing either the twist or envelope conformation. Therefore, its geometry was analysed using the procedure of Cremer & Pople (1975). For five-membered rings the geometry is fully described by a phase angle  $\varphi$ , which is an even multiple of  $18^{\circ}$  for the envelope conformation, an odd multiple of  $18^{\circ}$  for the twist conformation, and a total puckering amplitude,  $Q$ . For reference,  $Q = 0.353$  Å for the furanoid ring of sucrose. We find for the amidinium ion  $Q = 0.171$  Å and  $\varphi = 295^{\circ}$  [atom 1 is N(1), counting counter-clockwise in Fig. 3]. This implies that the largest contribution to the puckering of the ring is an envelope conformation, with the associated mirror passing through C(3), but that a twist conformation with the twofold axis passing through C(1) also contributes significantly to the puckering of the ring.

The central C–N bond lengths of the cation are essentially equivalent and are similar to the C–N bond lengths in acetamidinium chloride whose average length is 1.307 Å (Cannon, White & Willis, 1976). They are also similar to C–N bond lengths in the  $[H_2 \text{ malondiamidine}]^{2+}$  ion, whose average length is 1.304 Å (Pinkerton & Schwarzenbach, 1978). The C(1)–C(2) bond length in the cation reported here is similar to the equivalent bond length in the acetamidinium ion [1.477(3) Å] but is marginally shorter than the equivalent average bond length in the diprotonated malondiamidine ion [1.498(4) Å], although the environment of these latter bonds more closely approximates the environment of the C(1)–C(2) bond in the present study. The N–C–N angle is opened by the requirements for ring closure to  $125.7(3)^{\circ}$  in contrast to values near  $121^{\circ}$  in the aforementioned related ions. In compounds in which a C atom is bonded to three equivalent N atoms, such as in the guanidinium ion

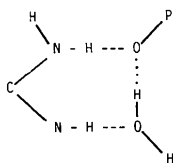
(Haas, Harris & Mills, 1965), the C–N distance is 1.323 Å.

This crystal structure contains stereochemical features which relate to protein–phosphate interaction of substituted guanidinium ions. These interactions have been discussed by Suwalsky (1968) and Lewin (1967, 1969). The crystal structures of three model systems for the interaction have been published, namely L-arginine phosphate monohydrate (Aoki, Nagamo & Itako, 1971; Saenger & Wagner, 1972), methylguanidinium dihydrogen orthophosphate (Cotton, Day, Hazen & Larsen, 1973) and propylguanidinium diethyl phosphate (Furberg & Solbakk, 1972). In the arginine complex there is no specific guanidinium–phosphate interaction in the complex hydrogen-bonding scheme, which also involves water, and the ammonium and carboxylate groups of arginine. In the latter two compounds, the cation and anion are directly doubly linked.



This interaction was postulated by Suwalsky (1968) to be the important one for arginine in biological systems.

Springs & Haake (1977) have shown that guanidinium ions produce a 27-fold rate enhancement for the cleavage of phosphodiester by the fluoride ion. They also showed that for bis(*p*-nitrophenyl) phosphate, the  $-\text{NH}_2^+$  groups of guanidinium chloride are only slightly more associated to the phosphate than are water molecules. Cotton, Hazen & Legg (1979) have resolved the structure of the staphylococcal nuclease–thymidine 3',5'-diphosphate complex to a resolution of 1.5 Å. They found that the direct double hydrogen bonding of two arginine residues to the phosphate O atoms plays an essential role in positioning the model substrate for nucleophilic attack by a water molecule. However, they also concluded that the water molecule in question is positioned for attack by first forming a hydrogen-bonded water bridge between a phosphate O atom and the carboxylate group of a glutamine residue. 'Water bridges' were postulated by Lewin (1967) as being involved in protein–phosphate interactions, particularly for the more moderately basic lysine residues, where the  $\epsilon$ -ammonium group is involved. In the compound whose structure is discussed here, we find hydrogen-bonded water bridges between the anion and the cation.



The fact that the relevant section of the cation in this study forms part of a five-membered ring would not seem to significantly alter its geometry from that of substituted guanidinium ions. The opening of the N–C–N angle to accommodate ring closure is compensated by a shortening of the C–N bonds. The basicity of the amidinium ions is lower than that of guanidinium ions. The water bridge occurs for the amidinium group but not for guanidinium itself. Therefore, it appears that the highly specific interaction of arginine with phosphate groups depends on two favorable conditions, the stereochemical capability of forming two collinear N–H...O hydrogen bonds, and a basicity that excludes water bridges.

In the phosphate ethyl group in which the H atoms were not located, the C–C bond length is unusually short and the thermal motion is very high. Difference syntheses of this region reveal residual electron density in the vicinity of the C atoms, indicating anomalous thermal motion or partial disorder. Similar effects have been reported for other diethyl phosphates (Hazel & Collin, 1972; Ezra & Collin, 1973).

The conformation of phosphate diesters has been of interest because of their role in determining nucleic-acid conformations. In particular, the literature contains numerous examples of structural determinations of oligonucleotides, many of which have been reviewed recently by Dickerson, Drew, Conner, Wing, Fratini & Kopka (1982). The O–P–O–C torsional angles in A-DNA, B-DNA and RNA polymers show these angles to have the *gauche*<sup>−</sup>, *gauche*<sup>−</sup> ( $P_3$ ) conformation (Kim, Berman, Seeman & Newton, 1973) although variations exist in these torsional angles. In the present compound both the *gauche*<sup>−</sup>, *gauche*<sup>−</sup> and *gauche*<sup>+</sup>, *gauche*<sup>+</sup> ( $A_1$ ) conformations are present because of the center of symmetry in the space group. The latter conformation is not consistent with polymerization of naturally occurring compounds, but does occur in some dinucleotides and trinucleotides such as uridylyl-3',5'-adenosine (Sussman, Seeman, Kim & Berman, 1972), and adenylyl-3',5'-adenylyl-3',5'-adenosine (Suck, Manor & Saenger, 1976).

The conformation of the diethyl phosphate anions in the barium (Kyogoku & Itaka, 1966), magnesium (Ezra & Collin, 1973) and propylguanidinium (Furberg & Solbakk, 1972) salts is similar to that found here. It is also similar for the ammonium salt of dimethyl phosphate (Giarda, Garbassi & Calcaterra, 1973) and has been predicted theoretically by Newton (1973). In contrast, silver diethyl phosphate has the *gauche.trans* conformation. The requirements for the formation of helices in naturally occurring polynucleotides is clearly not inconsistent with the preferred conformation of phosphodiester.

The torsional angles P–O(1)–C(7)–C(8) and P–O(2)–C(9)–C(10) are *trans*, with values of  $\pm 171.8$  and  $\pm 176.7^\circ$  respectively. Their usual values in naturally occurring polynucleotides are also *trans*.

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## Structure of 3-Benzyl-1,4,7,10,13-pentaoxacyclopentadecane-2,5,9-trione

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**Abstract.**  $C_{17}H_{20}O_8$ , monoclinic,  $P2_1/b$ ,  $Z = 4$ ,  $a = 10.798$  (11),  $b = 9.057$  (10),  $c = 17.585$  (31) Å,  $\gamma = 93.02$  (2)°,  $V = 1717.4$  Å<sup>3</sup>,  $D_x = 1.36$  Mg m<sup>-3</sup>,  $\mu = 0.106$  mm<sup>-1</sup>. The structure was solved by direct methods and refined by least-squares procedures to a final  $R$  of 0.048 for 2254 independent reflections. The interatomic distances and bond angles are in good agreement with previously published values. The 15-membered ring forms a distorted square. The three ester groups are in the *trans* form: two groups are almost perpendicular to the mean plane of the macrocycle and the third is displaced from it. The overall conformation is stabilized by both intra- and

intermolecular  $C=O \cdots C=O$  and  $C-H \cdots O$  (ester or ether) interactions.

**Introduction.** Publications on structures of macrocyclic molecules are devoted mainly to the cyclic polyethers (crown ethers) and their complexes (Dalley, 1978; Ovchinnikov, Ivanov & Shkrob, 1974). However, little information is presently available about crown-ether derivatives containing ester and amide groups.

We have previously reported (Ganin, Lukyanenko, Dvorkin, Popkov, Simonov & Bogatsky, 1981; Simonov *et al.*, 1981) the results of X-ray structure